Advanced Nanoporous Composite Materials for Industrial Heating Applications

Towards Low-Cost Nanostructured Refractory Materials

Arlon J. Hunt, LBNL
Siu-Chun Lee, ASL

E.O. Lawrence Berkeley National Laboratory

Applied Sciences Laboratory

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OUTLINE

- Programmatic Issues
- Technical Approach
- Progress in Material fabrication
- Thermal Testing results (Siu-Chun, ASL)
- Future Plans and Commercialization

Advanced Nanoporous Composite Materials for Industrial Heating Applications

Goal: Develop superior insulating and refractory materials for high temperature IPH applications.

Challenge: Fabricate new nanoporous materials using aerogel technology for improved cost/benefit.

Benefits: Reduced energy consumption by industrial furnaces and heat treatment equipment.

FY05 Activities: Process scale up, testing, and design - transfer technology.

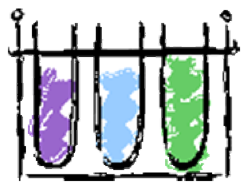
Participants: Lawrence Berkeley National Laboratory, Applied Sciences Laboratory, insulation/refractory manufacturer.

Advanced Nanoporous Composite Materials for Industrial Heating Applications

- **Barriers:**
  - Lack of low cost/high performance IPH thermal insulation material.
  - Scale-up nanostructured materials to commercial sized blocks.
  - Difficulty of high temperature conductivity measurements.
- **Pathways:**
  - Develop nanostructured insulating materials.
  - Find inexpensive sol-gel process using bulk chemicals
  - Produce powdered material and reform into blocks.
  - Increase strength of reformed blocks using fibers.
- **Metrics:**
  - Produce material with lower cost/R-value than existing insulation. Estimated energy savings 100 trillion BTU; cost savings $750 Million.

Sol-gel Processing for Nanostructured Materials

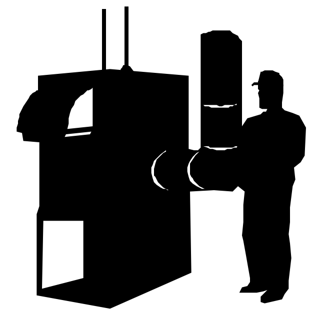
Combine
Chemicals



Allow solution
to gel

Supercritical
Drying of powder

Heat
processing



Pressing
& reheating

All five processes must be optimized

*** For properties**

*** For Scale-up**

Challenges for High Temperature Applications

- Must withstand temperatures of 700-1500 °C
  - Increase sintering resistance
- Must be chemically inert
- Must show reduced thermal conductivity
  - Target: 0.01-0.10 W/m-K
    - Block solid, gaseous, and ***Radiative*** heat transfer modes
- Must be affordable
  - Target: $5-20 per board foot

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Production Issues

- Many types of aerogels require lengthy synthesis, aging, and drying times
- Early Al_2O_3/ Cr_2O_3 aerogel preparations required 1-2 weeks of processing
  - Rapid gel time achievable with carefully controlled water content
  - Lengthy soaking time eliminated due to lower water content
  - Rapid supercritical drying practical for powdered aerogel production
- Total process time now reduced to ~1-2 days

Sol-Gel Synthesis Issues

- Large batches (~5 liter) require increased care to yield acceptable product
  - exothermic reaction requires good thermal control with large batches
  - Gel time varies considerably (up to 5,000X) with reactant concentrations
- Water content of sol found to be key to controlling gel time
  - low water gives precipitates, high water yields gel times >1 week

Prototype Block Preparation

- Various forming and firing processes evaluated to determine suitability for large-scale production
  - Wet-casting gave the best results
    - pure water adequate to cast various shapes
    - addition of binders was not necessary and has not provided significant added strength
    - minimal shrinkage or cracking
- 6 x 6 x 0.5” panel prepared for thermal testing
  - wet-cast into a stainless steel mold and fired at 1000 °C
- Composite structures using fibers should improve strength and durability
- Composition with fastest process leads to strongest blocks

Efforts identified best Composition and process

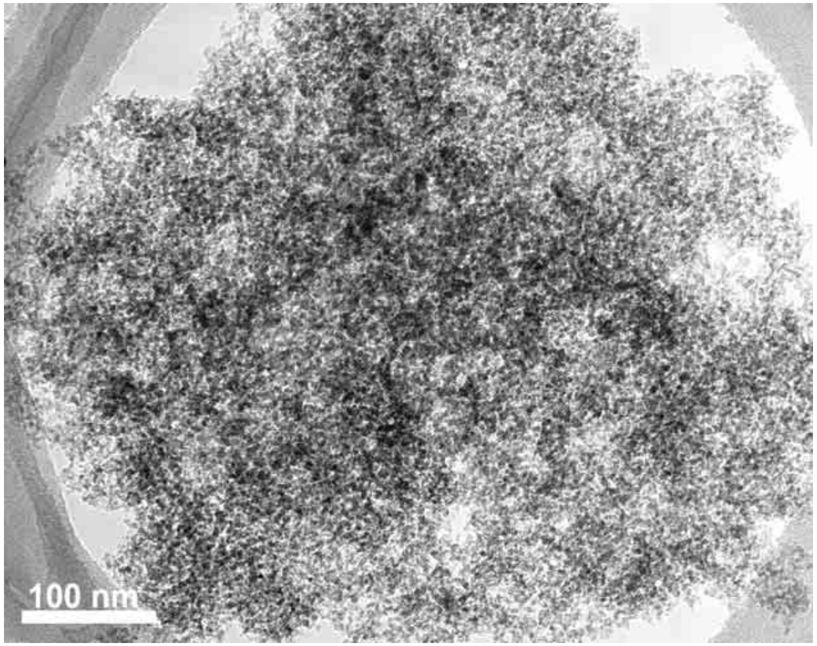
Thermal properties

Compound	Neat aerogel	450 °C	1000 °C
Cr_2O_3	290	13	13
$Al_2O_3 \cdot 2Cr_2O_3$	270	180	41
$Al_2O_3 \cdot Cr_2O_3$	260	160	44
$2Al_2O_3 \cdot Cr_2O_3$	240	170	64
$2(0.94Al_2O_3 \cdot 0.06SiO_2) \cdot Cr_2O_3$	350	—	130

Material Costs

Item	Cost/board foot ($)
Aluminum trihydroxide	0.23
CrO_3	1.36
Tetraethylorthosilicate	0.54
Al_2O_3 particulate opacifant	0.21
Total	**$2.34**

Electron Micrograph of Al2O3/ Cr2O3 /Si2O3



Alternative Chemistry Explored

- Other alternative compositions and processes examined include:
  - sol-gel syntheses using metal salts and epoxides (A. Gash, et al., ISA 6,7)
  - Additional redox-type reactions using various metal combinations
  - Sonochemical syntheses in liquid CO_2 of materials such as Boron Nitride
- None have matched the combination of physical properties and low cost shown by the Al_2O_3/ Cr_2O_3 aerogel powders

Thermal Performance

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- Thermal Testing
  - Equipment description
  - Thermal conductivity results
  - Comparisons with competitive materials
- Future Improvement
  - Performance enhancement by addition of fibers
  - Selection of fiber type and density will utilize validated analytical model

Heat Transfer Apparatus

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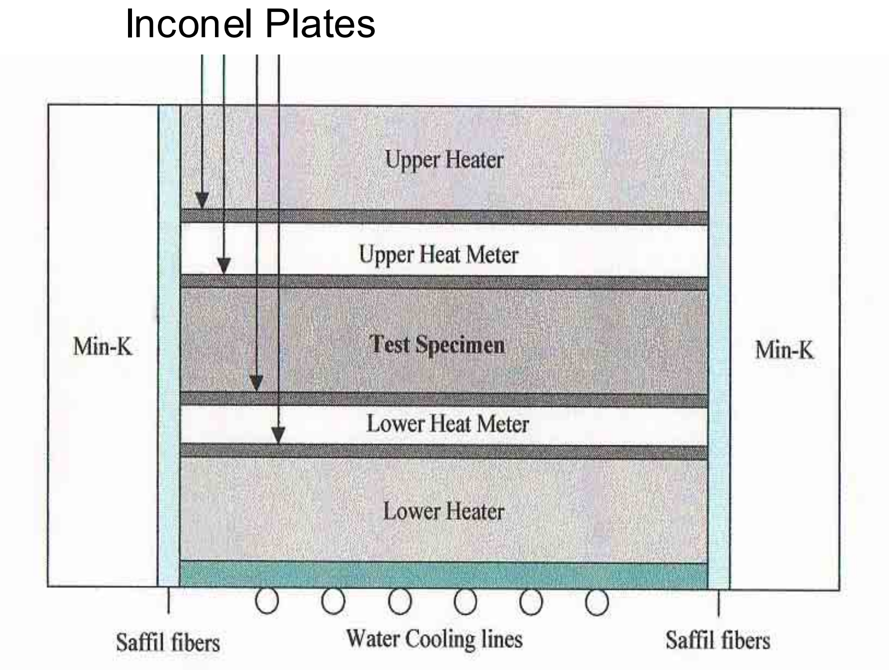
- 24” ID x 32”H stainless steel
- Ambient to 1E-7 torr

Heat meter apparatus:

- 300 to 1300K
- k: 0.005 to 1 W/m-K, ±10% uncertainty
- Specimen size: 15x15x2.5cm



Test Specimen

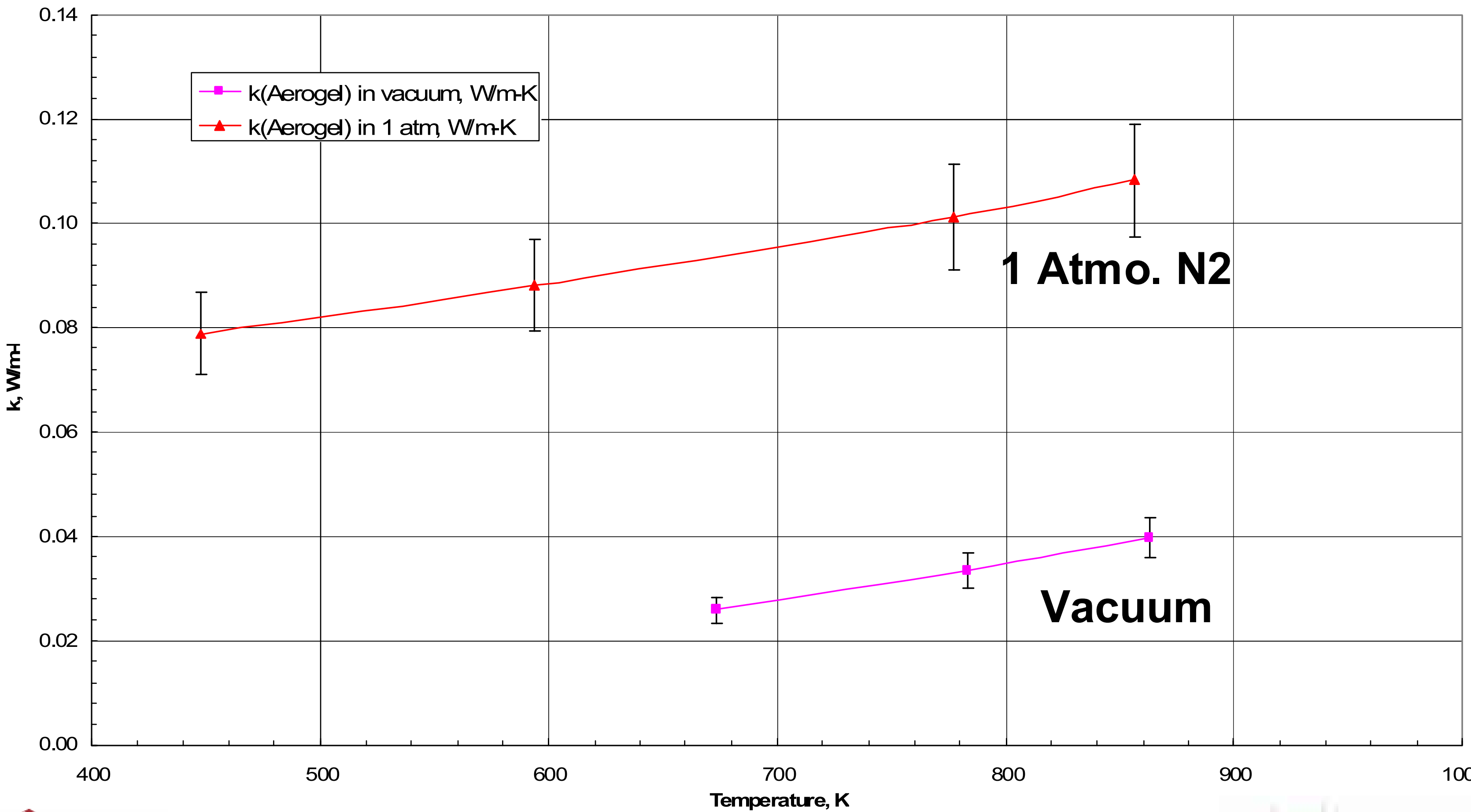
No apparent shrinkage due to testing from 400 to 900K.

- Tests initially in vacuum, followed by GN_2 at atmospheric pressure
- Pre-test specimen shows similar crack pattern.
- Cracking increased due to handling.

Specimen (post test)

15x15x1.05cm
888 kg/m^3

Measurements on Al_2O_3-Cr_2O_3 Aerogel **ASL**



Comparison with Competitive Materials

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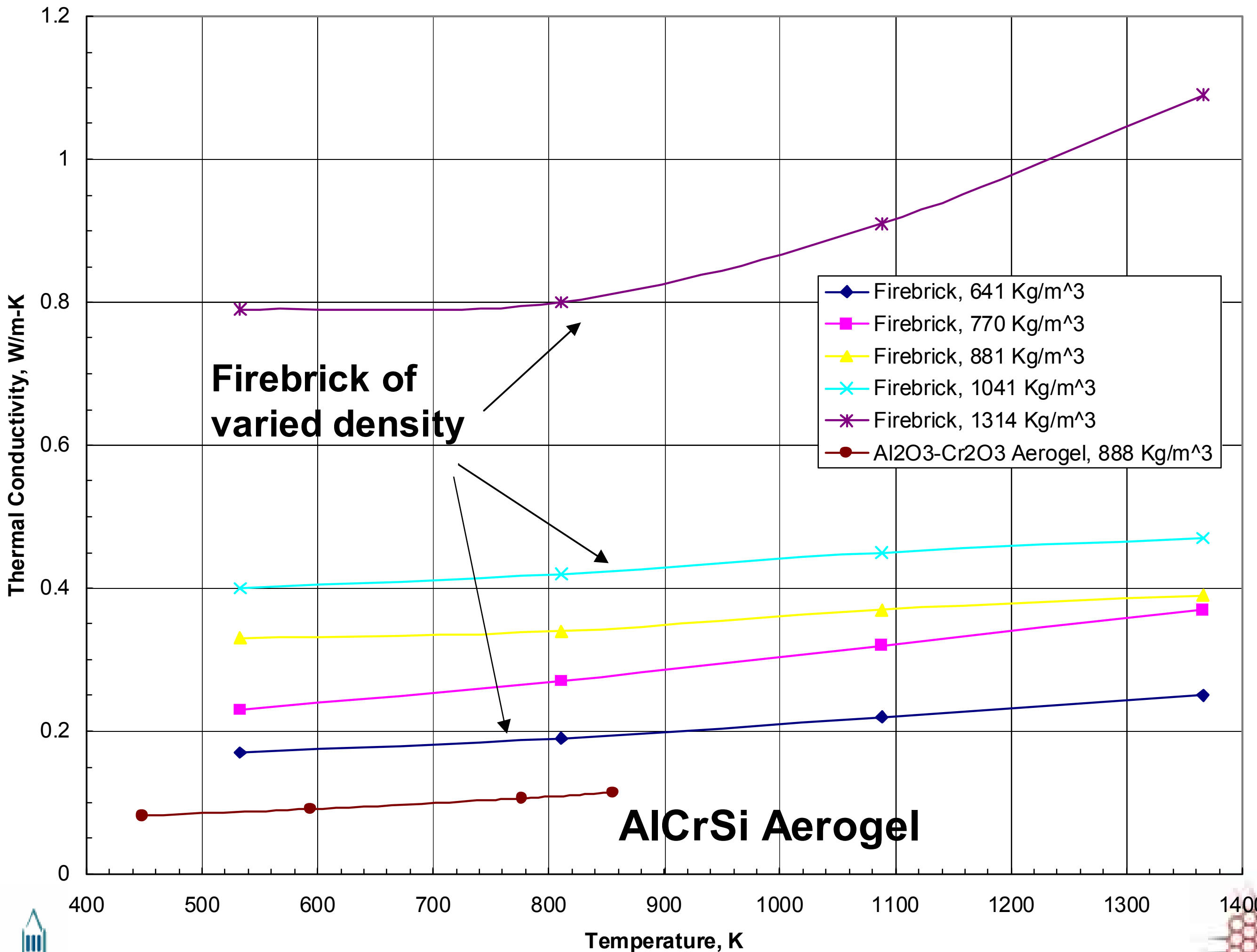

Comparison with LI900 Shuttle Tile Material

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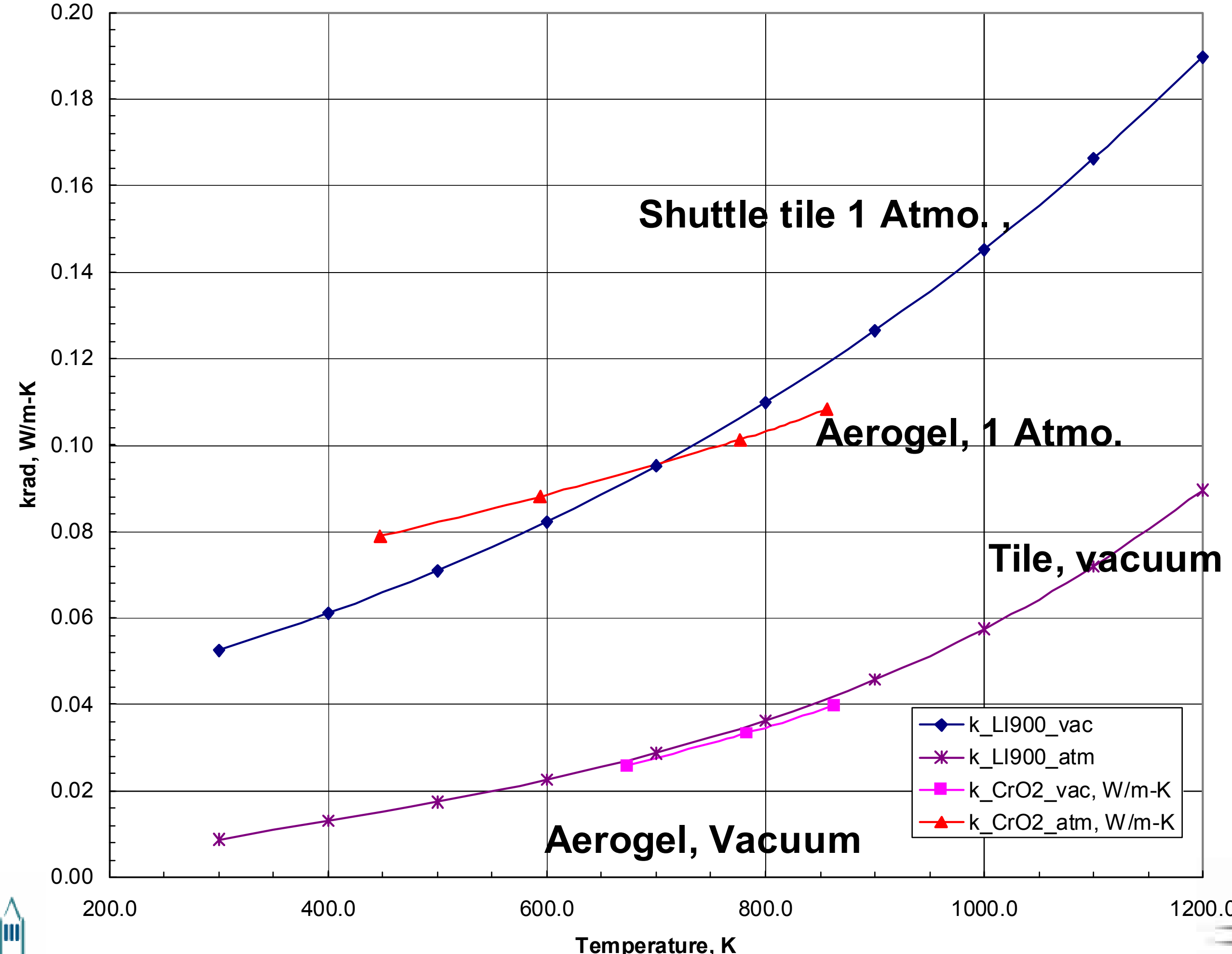

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Performance Enhancement by Fibers

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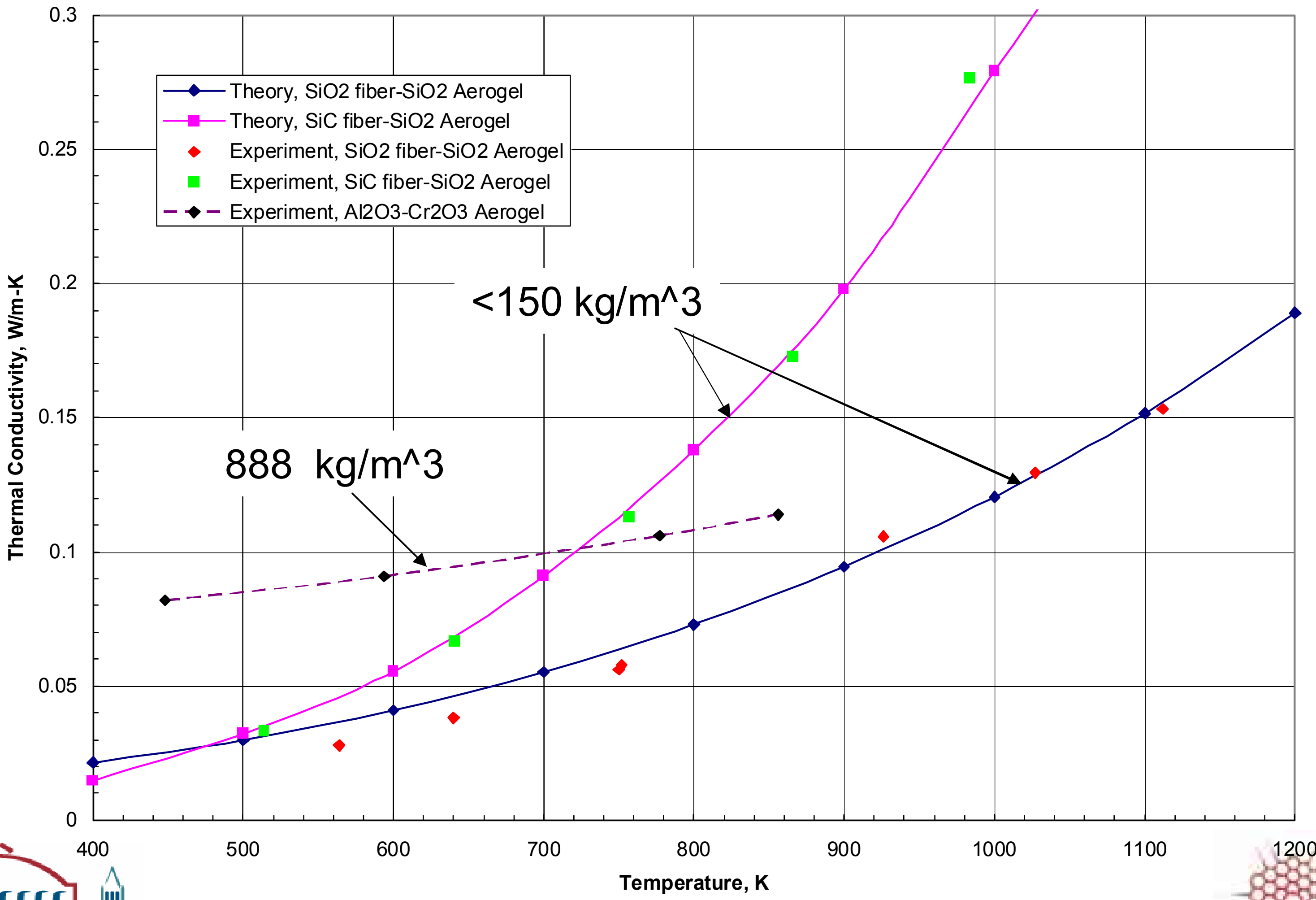

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Analytical Design Tool

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- ASL theoretical formalisms accurately predict heat transfer through composites of fibers and particulates.
  - Model has been validated by experimental data on various fiber composites, including fiber-filled aerogels
- Analytical tool will be used to achieve desired thermal performance by tailoring the material composition:
  - Material, diameter distribution, density, and orientation (fibers only) of fibers and particulates

Future Plans

- FY 05 goals:
  - Continue scale up activities
  - Improve strength and handle-ability by adding long fibers (alumina)
  - Optimize thermal performance by utilizing Applied Sciences Laboratory analytical design capability
    - Tailor the composition by optimal selection of density, fiber type, and fiber sizes
    - Facing treatments
  - Validate thermal performance by heat transfer measurements
  - Technology transfer to refractory/insulation manufacture